TITLE:	DETERMINATION OF HYDROGEN IN HIGH HEAT INPUT WELD METALS – AN UNDERSTANDING OF LRS & ISO: 3690 SPECIFICATION

CONTENTS:

1.0 SCOPE

The procedure described here is for measurement of diffusible hydrogen in the weld metal deposited by high heat input welding processes like, SAW, GMAW, FCAW, or similar processes.

2.0 METHOD

The method consists of using the welding consumable under test to make a sample weld bead on an assembly of steel pieces: one is the run-on piece, the middle one is the specimen from which the hydrogen level will be determined, and the third is the run-off piece. The pieces are held in a copper block, while the weld is being made, to provide rapid cooling. The welded sample is then released from the copper block & cooled to sub-zero temperatures to retain the hydrogen until the specimen can be cleaned, dried & submitted to analysis for hydrogen. The primary method of analysis uses a mercury filled gas burette to collect the hydrogen, which evolves from the specimen at near normal ambient temperature. The burette allows the direct measurement of the evolved volume. This can be related to the weight of deposited weld metal or the weight of the fused metal as required.

3.0 TEST SPECIMENS

Dimensions & Tolerances:

1. The length, width & thickness of the specimen blank depend on the welding process. The run-on & run-off pieces have the same width & thickness as the specimen blanks, but their lengths vary also according to the process. The dimensions are as follows:

Process	Test Specimen Blank (Middle piece)			Run-on piece	Run-off piece
	Length	Width	Thickness		
SAW	15	30	10	135	135
Note: All dimensions are in millimeters and Tolerance: ± 0.25 mm					

2. The longer run-on & run-off pieces are to allow a longer time for the larger bead welding conditions to stabilize before the weld pool moves onto the specimen blank piece.
3. The tolerance on all dimensions is ± 0.25 mm, but it is important that each set of three blank pieces (run-on, specimen & run-off) shall be ground as a set in order to ensure equally good contact with the fixture along the whole length of the assembly.
4. Before welding, each specimen blank is to be weighed to the nearest 10 milligram.

4.0 WELDING FIXTURES

The dimensions of the fixtures are shown in Figure 1. It consists of two water-cooled copper blocks with one block fixed and other one movable mounted on a steel plate. A copper foil (1.0mm thick) is placed between the steel blanks and the copper fixtures. These are of annealed (softened) copper so as to enable the best thermal contact with the fixture. These copper foils can be re-used but are to be annealed between each application. Annealing is to consist of heating to approx. 700°C and quenching immediately in water. The oxide formed during heating is then removed by pickling in dilute (10%) nitric acid followed by washing with distilled water and drying.

For SAW, the copper foil needs to extend above the upper face of the fixture (shown in Figure 2) to provide support for the flux during welding. The latter should help to reduce scatter in the values determined in the subsequent analysis of hydrogen content. In all cases the weld bead deposition is not to cause the copper foil to melt. If this happens, it means that either the weld bead is too wide, or the weld bead has not been deposited along the centerline of the steel blanks. If the weld bead is too wide (width almost same as that of the test piece), the welding conditions need to be adjusted to produce a narrower bead.

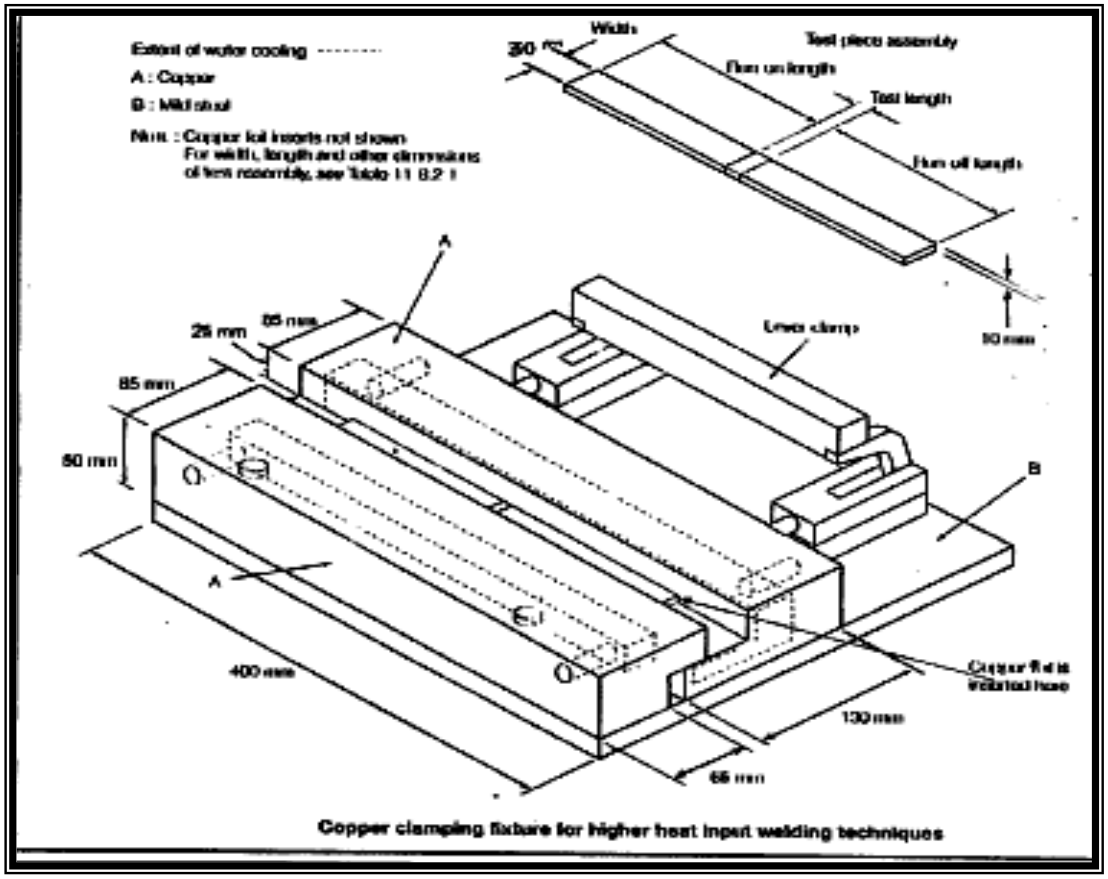

Figure 1: Copper clamping fixture

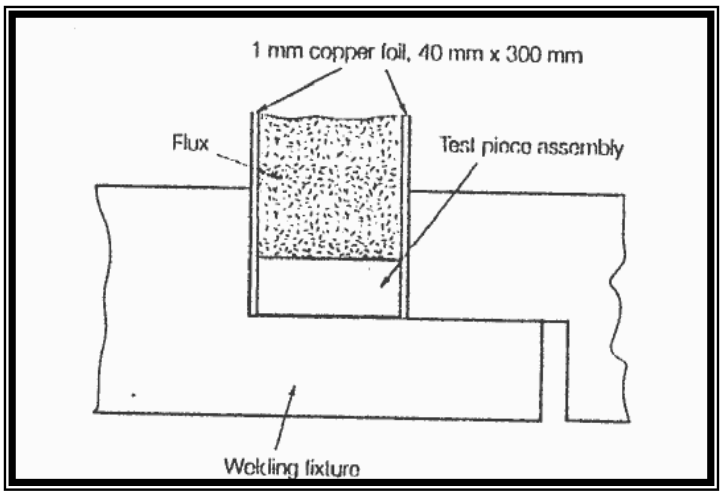

Figure 2: Copper foil extended for SAW technique

5.0 WELDING PROCEDURE

Target Conditions:

Process & Technique	Electrode diameter, mm	Welding current, amp	Average welding current, amp	Arc Voltage, V	Travel speed, mm/min	Max. heat input rate, KJ/mm	Test bead length, mm
SAW	2.50	270-350	300	28-30	300-500	3.0	200
	3.15	350-450	400	28-30	300-500	3.0	200
	4.0	450-550	500	28-30	300-500	3.0	200
GMAW	0.80	140-180	160	26-28	-	3.0	200
	1.20	160-240	200	26-28	-	3.0	200
	1.60	200-260	230	26-28	-	3.0	200
Heat input rate in KJ/mm = $\frac{\text{Arc voltage} \times \text{Welding current} \times 60}{\text{Travel speed (in mm/minute)} \times 10^3}$							

If the welding parameters need to be modified to avoid a bead width which melts the copper foil, the values shall be recorded together with the remark: “To avoid melting of foil”.

Test samples:

a) The fluxes for SAW should be re-dried as per the recommendations in either published literature or on the packing before making the test sample welds. These must not be dried at a higher temperature or for a longer time. If no recommendations are published or displayed, the temperature & time shall not exceed 350°C & two hours respectively.

b) The welded sample is to be released from the welding jig within 3.0 seconds after the arc is extinguished.

c) The sample is then quenched immediately in iced water followed by dipping in a bath of acetone or alcohol saturated with solid carbon dioxide. The temperature of the bath is maintained at about –75°C to avoid any loss of hydrogen from the welded test piece.

d) The pieces are to be wire brushed & broken apart while cold.

e) If the start of measurement of the hydrogen is to be delayed by more than an hour, it is recommended that the specimens are to be stored in liquid nitrogen to avoid any loss of hydrogen. If this is not possible, then within 3 days of welding the specimens must either:

- be inserted into the standard type of hydrogen collection mercury burette, or
- be analyzed for hydrogen in an accepted rapid analysis apparatus.

In either case, the test specimens are to be kept at the temperature of solid carbon dioxide while waiting to avoid any loss of hydrogen from the welded test piece.

6.0 ANALYSIS PROCEDURE

6.1 Handling & insertion of the specimen

When transferring the sample to gas burette, a shield of dry nitrogen shall be applied to avoid condensation of atmospheric humidity. The sequence of operation & the time spent in each shall be as follows:

a) The sample shall be washed in alcohol for a period of 3 to 5 seconds.

b) The sample shall be washed in pure ether for a period between 3 to 5 seconds.

c) The sample shall be dried in a blast of dry nitrogen supplied from a nozzle, particular attention being paid to the fractured surface of the specimen. The operation shall be accomplished in not less than 20 seconds & not more than 22 seconds.

d) While maintaining a blanket of dry nitrogen, the sample shall be transferred to the outer limb of the burette where the sample shall be held in position clear of the mercury surface by a magnet. The outer limb of the burette shall then be evacuated down to a pressure of approx. 13 Pa (0.1 mm Hg). The time spent in these operations shall be not less than 20 seconds & not more than 25 seconds.

e) The sample shall then be transferred through mercury air lock to its final position in the measuring limb of the burette. This operation shall be accomplished within 5 seconds.

f) The total time spent in transferring the sample until measurements commence shall not exceed 60 seconds.

6.2 Analysis Procedure

The sample shall be maintained at 25 ± 10 °C for a period of 72 hours when the final volume shall be measured. The barometric pressure as well as the precise temperature shall be recorded. The sample shall be removed from the apparatus & thoroughly brushed to remove any oxide skin & then weighed to an accuracy of 10 milligram.

7.0 CALCULATION AND EXPRESSION OF RESULTS

a) The volume V_h of diffusible hydrogen per 100 gm of deposited metal shall be calculated from the following formula:

$$V_h = \frac{V_g\ (B - H)}{760} \times \frac{273}{(273 + T_R)} \times \frac{100}{(M_2 - M_1)}$$

Where,

V_h = Volume of diffusible H_2 (ml/ 100 gm of deposited metal) at NTP (0°C & 760 mm Hg)

V_g = Volume of gas in burette in ml after 72 hours

B = Barometric pressure in mm Hg

T_R = Room temperature in °C when V_g is measured

H = Head of mercury in mm at which V_g is measured

M_2 = Mass of sample in gm after removal from apparatus

M_1 = Mass of sample in gm before deposition of weld

b) For the purpose of this procedure, an average value of hydrogen content (ml/ 100gm) obtained from at least 3 determinations shall be reported.

8.0 ASSESSMENT OF RESULTS

a) Qualification of H15, H10 & H5 for welding consumable approval is based primarily on V_h values of ≤ 15 cm^3, ≤ 10 cm^3, ≤5 cm^3 of hydrogen per 100 gm of deposited metal, respectively.